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Separation and Purification Technology 30 (2003) 229–239

Separation and Purification Technology

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Ultrafiltration: a means for decolorization of cane sugar solution

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Received 18 October 2001; received in revised form 4 August 2002; accepted 7 August 2002

Abstract

Membrane ultrafiltration was used for clarification as well as for decolorization of raw brown sugar obtained from the Indian sugar industry. Experimental results were obtained using sugar solutions of 28 and 46°Brix and mineral membranes of 20 nm, 5 and 1 kDa molecular weight cut-off (MWCO) on an industrial size pilot plant under different operating conditions. It was found that, even with the membrane of MWCO of 1 kDa, the maximum color removal was limited to 58.67% and steady-state permeate flux was only 29 1/h m^2 for the 46°Brix sugar solution. Empirical relationships between membrane performance (for decolorization and clarification) and membrane pore diameter were obtained from the experimental results. In order to obtain very high quality white crystalline sugar, further processing with adsorbents or use of an ion-exchange technique is required.
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Keywords: Cane sugar; Ultrafiltration; Color removal; Permeate flux; Membrane pore size

1. Introduction

The sugar industry is one of the largest food industries in India, with more than 500 sugar mills presently in operation [1]. The very old process consisted of heating the cane juice until solids are formed and that was called *Shakkara* (in Sanskrit). In a conventional process, the cane juices from different milling plants (normally 5) are combined and pumped to heaters and clarifying stations. The raw mixed juice obtained from 5 successive milling units contains sucrose and various impurities, such as reducing sugars, organic acids, amino acids, proteins, starch, gums, coloring matter, and other suspended matters. Bagasse goes directly to factory boilers and final molasses are sent to distillery. The mixed juice is treated at 98–105 °C with lime and sulfur dioxide, and then adjusted to a pH value of about 7. After sedimentation, the clear juice is pumped to evaporators to increase the °Brix up to 60–65. It is then sent to vacuum pans and raw sugar is obtained [2–6]. This type of sugar is not suitable for industrial use or for food and beverage production because it contains high

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PII: S1383-5866(02)00145-4

ash, turbidity and reducing sugars and also has a high sediment content. In order to obtain white sugar, raw brown sugar obtained from vacuum pans is conveyed to an affination station. The clarification is done by phosphatation/carbonation treatment and the decolorization is achieved by use of bone char, ion-exchange or their combination. Finally white sucrose crystals are obtained by evaporation and crystallization.

The sugar refineries themselves are undertaking most research and development work. Given that bone char and activated carbon are acceptable decolorizing agents, application of any alternative technique would have to be cost effective, easy to incorporate within existing systems and non-polluting. Membrane processes, like: MF/UF/NF (microfiltration/ultrafiltration/nanofiltration), ion-exchange or electrodialysis (ED) can be used for decolorization and removal of other impurities [7–12].

Recently, clarification and decolorization by UF have gained importance and its use in the food industry is widely accepted because of its low energy consumption and simplicity of operation. Clarification by UF of sugar solution is proven to be technically superior to ordinary lime treatment because it yields a juice of higher purity and better color quality and is free from starch and acidified substances [7,13,14]. In a recent study, Karode et al. [13] examined, UF and coagulation coupled with UF for clarification/decolorization of raw brown sugar from the Indian sugar industry. Polyethersulphone (PES) membranes (5–100 kDa MWCO) and mineral Carbosep membranes (15–50 kDa MWCO) were used for decolorization. With both PES and mineral membranes of molecular weight cut-off (MWCO) between 30 and 50 kDa, approximately 50% reduction in color was obtained. It was found that UF yields, a juice of great purity and better quality, and was a viable option for final decolorization and crystallization.

Ghosh et al. [6] used organic membrane modules of 20 kDa MWCO in spiral form for treating raw and clarified juice. Effects of the transmembrane pressure (TMP), operating time and feed temperature on permeate flux and quality were examined. The permeate obtained was found to be consistently better in quality (in terms of higher clarity, lower color and reduced calcium oxide content) than the clarified juice produced by the conventional double sulphitation process. The authors did not report any results on color reduction although it was mentioned that the juice color changed from brown to yellow after UF. These results confirmed that, membranes with pore sizes between 10 and 30 kDa MWCO were suitable for sugar solution clarification.

Raw cane sugar (or brown sugar) normally contains 94–98.5% sucrose and 1.5–6% non-sucrose components of various types. Polysaccharides and different colorants are mainly responsible for the quality of refined sugar after crystallization. [7,15]. The colorants in raw sugar can be of two types: natural colors (flavonoids, chlorophylls etc.) present in cane and other colors which are formed during further processing (melanoidins, caramels and from degradation of reducing sugars etc.). Natural colors are characterized by their low molecular mass (<1 kg/mol) and are mostly eliminated during purification. The retained colors after purification are of high molecular mass (from 1 to 2000 kg/mol) and need to be removed in the preparation of white sugar crystals. Decloux et al. [7] demonstrated that in order to obtain reliable decolorization using UF, precautions must be exercised in color analysis and in application of operating conditions. They concluded that temperature was the most important factor and best decolorization was obtained at 60 °C, with a 3 bar TMP and 2.5 m/s cross flow velocity using a mineral membrane (Kerasep) of 15 kDa MWCO.

A new Applexion process was integrated in a sugar mill [16] in which the solution of brown sugar was decolorized with an ion-exchanger, then crystallized in single or double-stage crystallization [8,9]. The ion-exchange resins removed color from cane sugar liquor and NF was found suitable for recovering sodium chloride and removing organic matter from the regeneration effluent. Cartier et al. [8] reported colorant removal of up to 99% and sodium chloride recovery in the permeate of up to 90%.

ED can also be used for the removal of inorganic matter from clarified sugarcane juice [17,18]. Although some minerals such as phosphates, silica and magnesia are partially removed

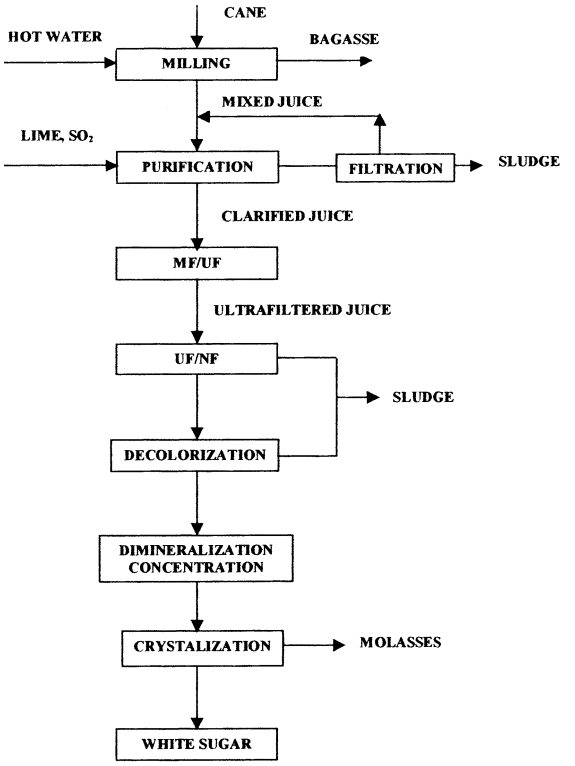


Fig. 1. Modified membrane process for cane juice treatment.

by clarification, potassium, sodium and low concentrations of sulfates are not completely removable. These minerals tend to be concentrated further during processing. There are also several other compounds like organic acids, amino acids, and enzymes that are present in the juice as impurities. With the ED process 50–70% of salts can be removed and the purity of sugar crystals obtained can be up to 99%.

In most of those studies, the membrane process was applied for improving the quality of raw brown sugar, i.e. for removing colorants. This type of treatment has been proved to be economical to produce fewer pollutants and to yield sugar with better quality ($IU < 200$) than the sugar obtained from with the conventional method ($IU > 300$).

Fig. 1 presents a modified membrane process, where the treatment using the membrane operation starts with fresh cane juice. The purification step (liming and sulphitation) is required for removing suspended matter and other impurities. The purified juice is processed by MF, then by UF and finally by NF (the NF is also not necessary, if the color removal at UF level is acceptable). Complete decolorization of the filtered juice can be obtained by ion-exchange. The decolorized juice is finally concentrated by evaporation and then crystallized to give white sugar crystals of high quality. At present, this type of cane sugar production process is not used. This may be because of very large fraction of the fresh cane juice is available only during 2–3 months, and it should be processed at a very fast rate in order to avoid the deterioration of juice quality. Therefore, the application of modified processes will need very large membrane surface areas (in other words high initial investment) but it can avoid the use of very large quantities of chemicals, and so will also produce a much smaller quantity of pollutants.

Most of the previous studies on clarification and decolorization of brown sugar solutions were made using organic membranes of different configurations and MWCO [19]. In contrast relatively few published experimental results are available on the use of mineral membranes for decolorization of raw sugar. In this work, we present UF results of raw sugar solutions from an industrial size pilot plant using various Membralox (SCT) mineral membranes. Results on reduction in color and permeate flux obtained at different operating conditions with membranes of different pore sizes are presented. Based on these results empirical correlations for membrane performance as a function of membrane pore diameter are developed.

2. Experiment

Experiments were conducted with raw Indian sugar solutions of 28 and 46°Brix and mineral membranes of different pore sizes under different operating conditions on an industrial size pilot plant designed and constructed by Polymem, France (Fig. 2). The experimental procedure may be described as follows. Raw sugar solution (contained in the double envelope feed tank R) was continuously agitated in order to maintain its

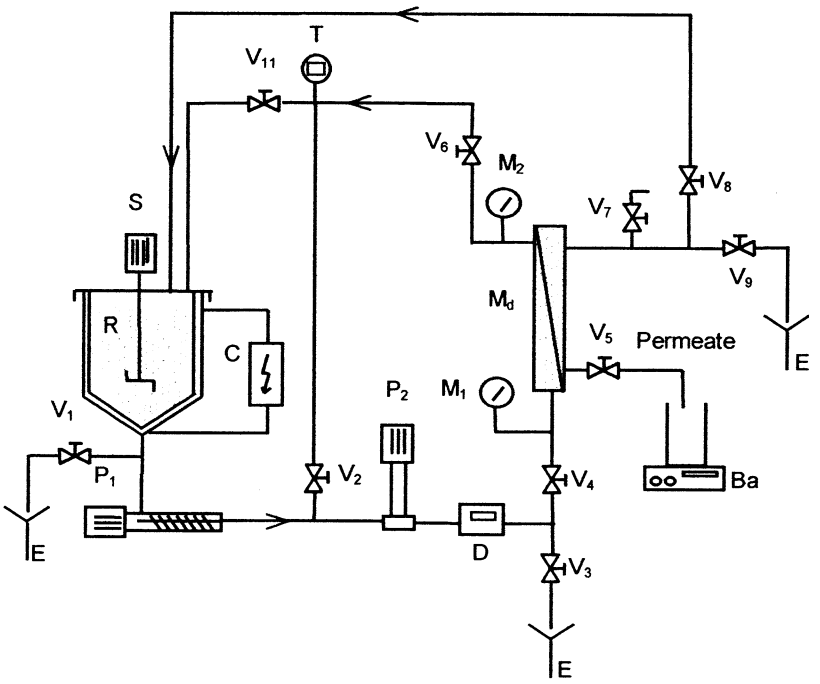


Fig. 2. Schematic diagram of the pilot plant (S, stirrer; B_a, balance; C, heating system; D, electromagnetic flow meter; E, drainage; M_1, M_2, pressure gauge; M_d, membrane module; P_1, P_2, pumps; R, feed tank; T, temperature probe; V_1–V_{11}, control valves).

homogeneity and was pumped with a gear pump P_1 to a Membralox membrane module at a specified cross flow velocity (U) and a constant TMP. Two valves, V_{11} and V_6 were used to control the TMP. Valves V_2 and V_4 and a centrifugal pump, P_2, were used to control the cross flow velocity. With the heating system, C, the solution temperature was kept at 90 ± 2 °C. The permeate flux was measured by weighing filtrate collected over a given time using a Sartorius balance, B_a, and the results together with other experimental data were sent to a monitoring system. Membralox membrane modules used in this study were purchased from SCT-U.S. Filter, France (module number IP19-40 1020 mm EPDM). Each membrane module is made of 19 circular channels (4 mm inner diameter), and has a total surface area of 0.243 m^2. Three different modules with pore size of 20 nm, 5 and 1 kDa MWCO were used in this study.

The sample cane sugar solution (~ 30 l) was prepared by dissolving correct quantity of raw brown sugar in demineralised water outside the feed tank, R and was pre-filtered on a glass fiber filter (>100 μm) in order to eliminate all the suspended materials, which could damage the pumps and block the flow circuit. Initial color concentration of sugar solution was measured as a reference. Experiments were conducted at TMP of 3 bar and 5 bar and at cross flow velocities of 5.4 and 7.7 m/s using 28 and 46°Brix solutions. After each experiment, the membrane was regenerated (cleaned) according to the following protocol: rinsing with hot water at 60 °C for 30 min; cleaning with a solution of Ultrasil 13 (2%) at 70 °C for 30–45 min; rinsing with hot water again at 60 °C for 30 min and verification of cleaning by measuring the water flux. With this protocol the membrane was found completely generated. The color concentration was determined by measuring the solution's optical density at 560 nm with a HACH DR/2010 spectrophotometer in accordance with the ICUMSA method [20]. That is, the solution sample (feed, retentate or permeate) was diluted to 5°Brix, the pH adjusted to 7 by adding NaOH, then filtered using a 0.45 μm

Table 1
Steady-state permeate flux and color reduction values at different temperatures

°Brix	TMP (bar)	U (m/s)	Temperature (°C)	J_{steady} (l/h m^2)	Decolorization (%)
			30	18	38.82
28	3	5.4	50	35	37.45
			90	73	39.28

Membrane, 5 kDa.

Table 2
Reproducibility of experiments

°Brix	TMP (bar)	U (m/s)	J_{steady} (l/h m^2)	Decolorization (%)
			133	23.80
46	5	7.7	126	27.56
			129	24.17

Membrane, 20 nm; Temperature, 90 °C.

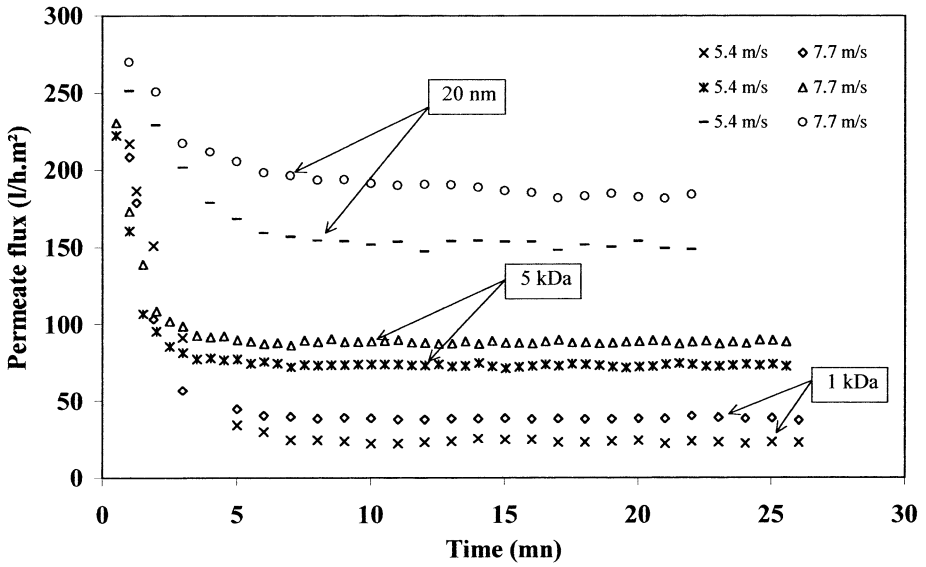


Fig. 3. Variation of permeate flux with time for two different cross flow velocities and at a constant TMP (°Brix = 28, TMP = 3 bar, T = 90 °C).

membrane and the color intensity of the treated solution was measured at 560 nm. Generally speaking, for color intensity determination, absorbance measurements are commonly made at 420 or 560 nm. Mak [21] reported that, for dark solutions, absorbance measurements at 420 nm were not optimal and marked improvement in sensitivity was obtained when transmittance readings were obtained at 560 nm. As the raw cane sugar solution used in this study was quite dark in color, measurements at 560 nm wavelengths were made.

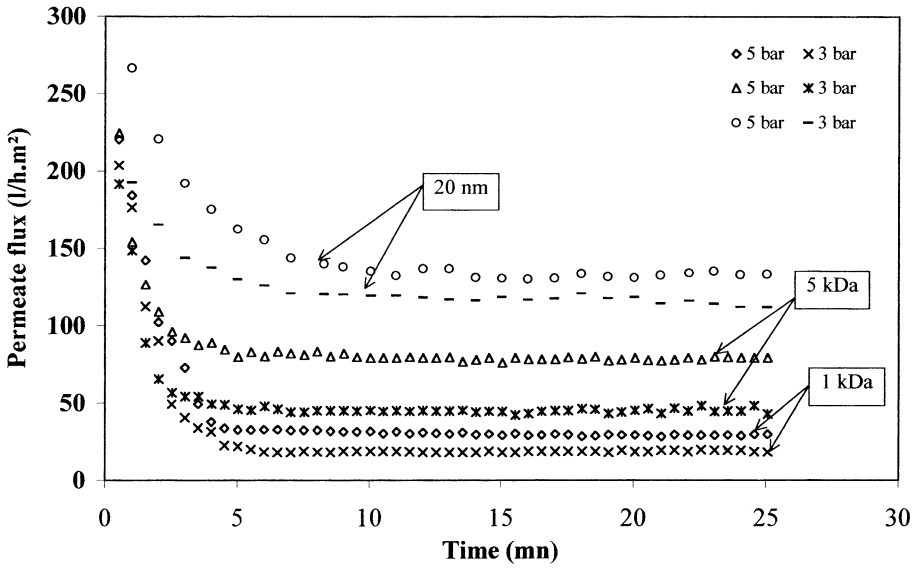


Fig. 4. Variation of permeate flux with time for two different TMPs and at a constant cross flow velocity (°Brix = 46, $U = 7.7$ m/s, $T = 90$ °C).

3. Results and discussion

3.1. Effect of temperature

In order to examine the importance of the feed solution temperature to color removal and permeate flux, measurements were made with sugar solutions (30 l) of 28°Brix at a TMP of 3 bar and a cross flow velocity of 5.4 m/s. The temperature of the feed solution varied from 30 to 90 °C. The results obtained after 2 h of operation are given in Table 1. Temperature was found not to have any appreciable effect on decolorization but the filtrate flux increased with the increase of temperature (~ 1 l/h m^2/°C). The small variations in decolorization may be simply due to errors in absorbency measurements.

3.2. Effect of the operating variables

For the study of the effects of the TMP and cross flow velocity on color reduction and permeate flux, we conducted 3 experiments using 46°Brix solutions and the 20 nm pore size membrane. The temperature of the sugar solution was fixed at 90 °C (about the maximum temperature applied in industry for decolorization) in order to avoid increased evaporation. Also, the permeate flux obtained at this temperature was maximum. The TMP was fixed at 5 bar and the cross flow velocity at 7.7 m/s. The steady-state permeate flux (J_{steady}) and the decolorization percentage [(color concentration in retentate—color concentration in permeate)/color concentration in retentate] are presented in Table 2. The measurement error of the permeate flux was of the order of 2% and that for decolorization was about 1%. It can be seen that both the reduction in color and the steady-state permeate flux remained essentially the same, confirming the reproducibility of our experimental work. The evolution of the permeate flux is shown in Figs. 3 and 4, for 3 membranes with different pore sizes and sugar solution of two different concentrations. In all cases, an initial fast permeate flux decline was observed. This drop was faster for membranes with MWCO of 1 and 5 kDa, i.e. a rapid fouling (by the rejected organic and inorganic particles on the membrane surface) followed by a slow stabilized (steady-state) permeate flux. The stabilized permeate flux was obtained after a period of about 5 min for the 1 and 5 kDa membranes, whereas for the membrane of 20 nm

Table 3
Steady-state permeate flux and decolorization values for different membranes

			Membrane, 20 nm		Membrane, 5 kDa		Membrane, 1 kDa	
°Brix	TMP (bar)	U (m/s)	J_{steady} (1/h m^2)	Decolorization (%)	J_{steady} (1/h m^2)	Decolorization (%)	J_{steady} (1/h m^2)	Decolorization (%)
28	3	5.4	148	23.34	73	39.28	23	54.16
		7.7	183	24.51	88	36.60	37	52.89
	5	5.4	198	25.42	101	37.62	48	53.63
		7.7	217	22.82	124	38.01	71	55.78
46	3	5.4	94	25.17	36	37.02	11	55.47
		7.7	112	24.37	43	35.64	18	56.12
	5	5.4	119	26.08	69	36.67	21	53.91
		7.7	133	23.80	79	37.48	29	58.67

size, this period was about 10 min. It was also observed that the permeate flux increased with increase in TMP and cross flow velocity and so this type of fouling could be of a reversible type. The long-term experiments ($\sim$2 h) show that the permeate flux remains constant and a drop of about 2 l of permeate volume per hour was recorded.

3.3. Permeate flux and decolorization

Table 3 presents percentage reduction in color and variation in the steady-state permeate flux value obtained from 2 types of sugar solutions, from different experimenta conditions and for different sizes of membranes (Figs. 3 and 4). It was found that the color reduction increased with the decrease in membrane pore size, however, for a given membrane pore size, the variations of TMP and the cross flow velocity do not have an appreciable effect on the color removal, which indicated that the removal of color components is simply a pore size exclusion. The permeate flux values on the other hand decreased with the decrease in membrane pore size and for a given pore size it increased with increase in TMP and cross flow velocity.

The percent decolorization with 20 nm membrane was from 22.82 to 26.08%, with 5 kDa from 35.64 to 39.28% and with 1 kDa it was from 52.89 to 58.67%. These results confirm that the percent decolorization increases with the decrease in membrane pore size. It is possible to obtain higher decolorization ($>50\%$) by using the membranes of pore size lower than 1 kDa, but the permeate flux would be less than 30 l/h m^2.

3.4. Effect of membrane pore size on decolorization and permeate flux

We attempted to establish empirical relationships between membrane performance and membrane pore diameter based on our experimental results. To convert the MWCO values to the corresponding pore diameter, we used the approximate expression: $\mathrm{MWCO} = 30\, d_{pm}^{5/3}$ where MWCO is the molecular weight cut-off in Dalton and d_{pm} is the membrane pore diameter in Å. We found that 1 and 5 kDa correspond approximately to an average pore diameter of about 0.001 μm and 0.002 μm. The membrane manufacturer (SCT) [22] confirms that the real mean pore diameter could be about 0.002 μm for 1 kDa and 0.004 μm for 5 kDa membrane module without providing any justification. We have used the calculated values of membrane pore diameter d_{pm} (obtained from the equation $\mathrm{MWCO} = 30\ d_{pm}^{5/3}$) for developing the empirical relationships for steady-state permeate flux and decolorization percentage variation with membrane pore diameter. The results are expressed as:

$$J_{steady} = A_1 d_{pm}^{\alpha}$$

$$\mathrm{Deco} = A_2 d_{pm}^{-\gamma}$$

Table 4
Empirical values of A_1, A_2, α and γ for the three membranes tested

		46°Brix solution				28°Brix Solution			
TMP (bar)		3		5		3		5	
U (m/s)		5.4	7.7	5.4	7.7	5.4	7.7	5.4	7.7
J_{steady}	A_1	127.3	1072.5	914.96	807.11	1368.2	1280.50	1118.60	856.99
	α	0.64	0.56	0.49	0.43	0.54	0.48	0.43	0.34
	R^2	0.87	0.93	0.76	0.79	0.82	0.88	0.89	0.91
Deco	A_2	9.55	8.83	10.75	7.84	8.06	9.48	10.10	7.47
	γ	0.24	0.25	0.22	0.27	0.27	0.24	0.23	0.28
	R^2	0.90	0.88	0.89	0.91	0.97	0.92	0.93	0.95

Experimental results were used to determine the values of the coefficients and exponents of these empirical relationships. The values of the coefficients, A_1 and A_2 and exponents, α and γ and the correlation coefficients R^2 are given in Table 4. Fig. 5 presents the variations of permeate flux and

Table 5
Experimental results collected from different publications

Membrane	Size	J_{steady} (l/h m^2)	Deco (%)	J_{floc} (l/h m^2)	$Deco_{floc}$ (%)	T (°C)	°Brix
Kerasep Techsep [23]	0.1 μm	50.0	23.0	40.0	45.0	85	50
	300 kDa	65.0	20.0	65.0	50.0		
	15 kDa	25.0	39.0	25.0	58.0		
Kerasep Techsep [7]	15 kDa	47.0	52.8	–	–	60	30
		114.0	65.4			80	
Carbosep Techsep [13]	15 kDa	8.0	88.0	40	55	70	50
	30 kDa	18.0	56.0				
	50 kDa	50.0	55.0				
Carbosep Techsep [24]	50 kDa	30.0	55.0	–	–	90	20
	300 kDa	70.0	32.0				
	0.14 μm	230.0	45.0				
	0.2 μm	260.0	38.0				
	0.45 μm	150.0	40.0				
Membralox SCT [10]	0.1 μm	38.0	47.0	–	–	80–90	60
.	0.2 μm	27.0	45.0				
	0.5 μm	30.0	46.0				
	0.8 μm	52.0	39.0				
	1.4 μm	62.7	27.0				
Membralox SCT	1 kDa	23.0	54.0	–	–	90	28
	5 kDa	73.0	39.0				
	20 nm	148.0	23.3				
Present results	1 kDa	11.0	55.4	–	–		46
	5 kDa	36.0	37.02				
	20 nm	94.0	25.17				

J_{steady}, steady-state permeate flux; J_{floc}, permeate flux with flocculation; Deco, decolorization; $Deco_{floc}$, decolorization with flocculation. Membralox SCT, present results.

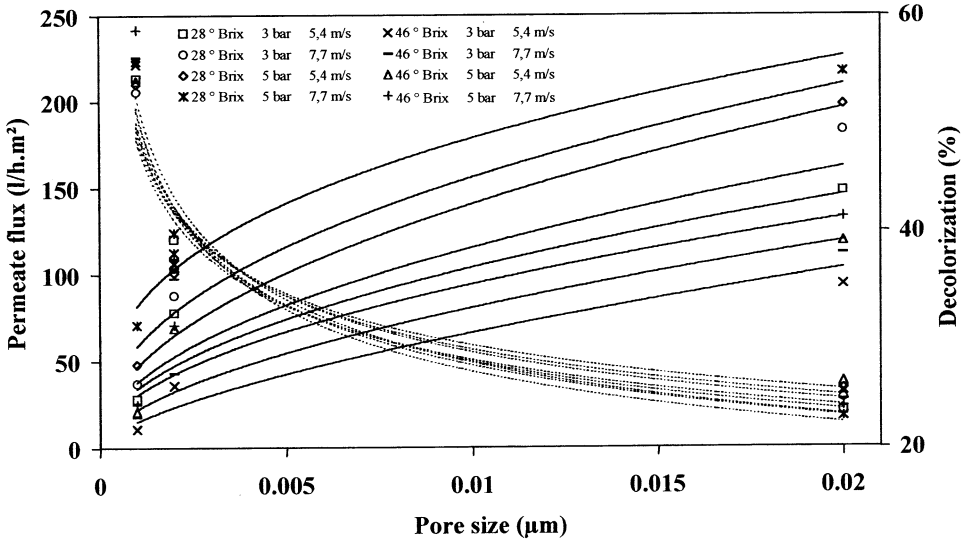


Fig. 5. Variation of percent decolorization and permeate flux with membrane pore size (28 and 46°Brix, $T = 90$ °C).

decolorization percentage with membrane pore diameter expressed in µm for two types of sugar-solutions.

The empirical relations obtained for 46°Brix sugar solutions (at TMP, 3–5 bar, $U = 5.4$–7.7 m/s and $T = 90$ °C with three different membranes) are given as: $\text{Deco} = 9.1863\ d_{\text{pm}}^{-0.2458}$ ($R^2 = 0.892$) and $J_{\text{steady}} = 1001.7\ d_{\text{pm}}^{0.533}$ ($R^2 = 0.738$).

4. Discussions

Normally raw cane sugar crystals obtained from the conventional process are colored and strongly aromatic because of the presence of many impurities. The use of a membrane process seems to be an efficient method for removing the color substances and simplifying further refining. The sugar content of membrane permeates remains the same as that of the feed solution since sucrose is not retained by the membrane.

In order to make some conclusions and compare the results, obtained on permeate flux and reduction in color with mineral membranes, we looked in to the published results from different researchers. It was not easy to collect published values for the same operating conditions, for the same type of membrane and for the same concentration of sugar solutions. Table 5 lists experimental results from different published literature and also from the present study. A few common parameters (membrane pore diameter, steady-state permeate flux, percent decolorization and about the same hydrodynamic conditions) have been selected for comparison.

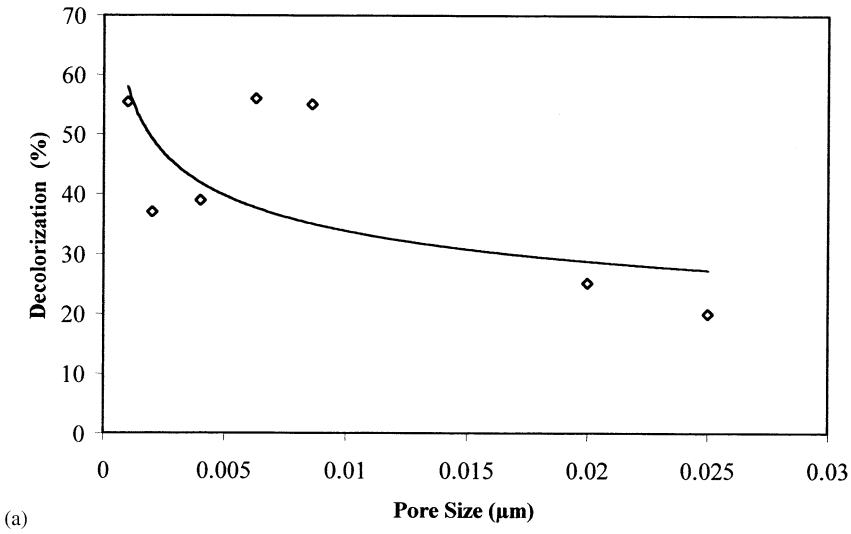
Experimental results from Cartier et al. [23], Karode et al. [13] without flocculation and from the present study were selected for developing empirical relations, as in these experiments the sugar solution used was of about 50°Brix, the temperature of feed was more than 70 °C and the cross flow velocity and TMPs were normally high. Fig. 6a and b present the percent decolorization and permeate flux variations with membrane pore diameter. The approximate empirical relations were to be:

$$\text{Deco} = 11.529\ d_{\text{pm}}^{-0.4424} (R^2 = 0.4424)$$

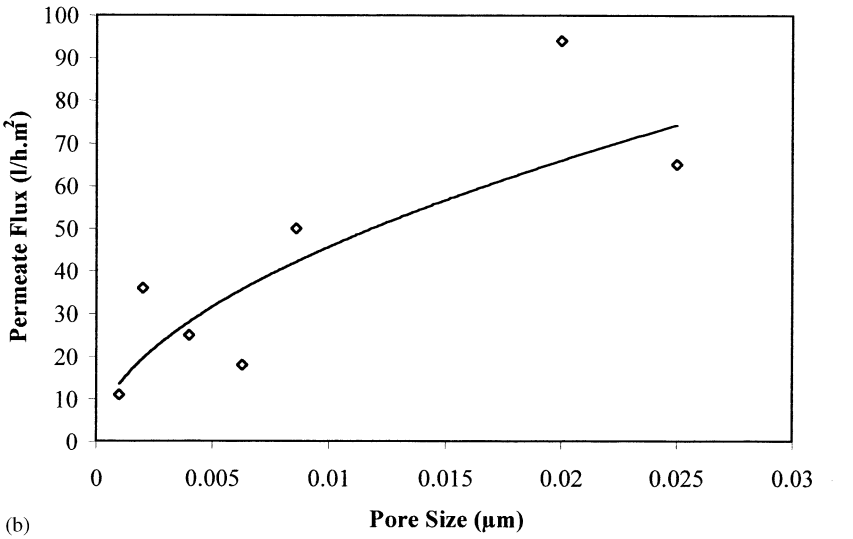
and

$$J_{\text{steady}} = 524.81\ d_{\text{pm}}^{-0.5301} (R^2 = 0.6814).$$

These relations can be used to predict approximate values of decolorization percentage and permeate flux with given membrane pore diameter. More accurate relationships can be obtained by



(a)



(b)

Fig. 6. (a) Variation of reduction in color with membrane pore size from Refs. [23,13] and present results for the same TMP and cross-flow velocity, (b) Variation of permeate flux with membrane pore size from Refs. [23,13] and present results for the same TMP and cross-flow velocity.

incorporating the other variables like: TMP, U and T.

5. Conclusions

From these experimental results it is clear that percentage decolorization of raw brown sugar solution increases and permeate flux decrease with the decrease in membrane pore diameter. Temperature was found not to have any appreciable effect on decolorization but the permeate flux increased with the increase of temperature. It was also found that for a given membrane pore diameter, the changes of TMP and the cross flow velocity values do not have appreciable effect on

the color removal. This indicates that the removal of color components is simply a pore size exclusion effect. The steady-state permeate flux values on the other hand decreased with the decrease in membrane pore diameter, and for a given pore diameter, it increased with increase in TMP and cross flow velocity. Mineral membranes of MWCO up to 1 kDa (nearly in the NF range) were used in this study and it was found that even with the membrane of MWCO of 1 kDa, the maximum color removal limited to 58.67%. These experimental results, therefore, confirm the presence of very small coloring molecules in the raw brown sugar, which are not retained by the membrane and pass into the permeate. It was concluded that further processing of the permeate (partially decolorized sugar solution) on absorbents (like bone char) or by ion-exchange resins is necessary for complete decolorization. Empirical relationships between membrane performance and membrane pore diameter can be used for predicting performance for this application.

Acknowledgements

The facilities for carrying out this research work were provided under project No. 1815-1 by CEFIPRA/IFCPAR (Indo-French Center for the Promotion of Advanced Research).

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